PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improved Process of Distillation of Linseed and similar Oils

We, EASTMAN KODAK COMPANY, a Company organised under the Laws of the State of New York, United States of America, of 343, State Street, Rochester, New York, United States of America, (Assignees of KENNETH CLAUDE DEVEREUX HICKMAN, a British Subject, of Kodak Park, Rochester, County of Monroe, State of New York, United States of America), do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to an improved process of distillation of linseed and similar oils.

It is known that the efficacy of a drying oil depends upon its ability to oxidise (or polymerise) so as to form a resinous skin or film. Thus, linseed oil when mixed with white lead so as to form paint, gradually oxidises on exposure to the atmosphere or forms a protective film of resinous material upon the painted surface. It has not previously been possible to obtain purified drying oils having a high degree of unsaturation, owing to difficulties in distilling such high boiling substances as naturally occurring vegetable and animal oils.

According to the present invention, unbroken linseed and similar oils are submitted to a process of molecular distillation and a "late" distillate fraction constituting a highly purified oil is collected, which is more unsaturated than the initial material. Molecular distillation is a specific form of high vacuum-short path distillation in which the distance separating distilling and condensing surfaces is equal to or less than the mean free path of the molecules of the distilland. It has already been proposed to submit animal oils to molecular distillation, so as to obtain vitamin concentrates but in this process, the vitamins distil in the early stages before any substantial distillation of unsaturated glyceride has taken place.

The high vacuum-short path distillation of oils which have first been "broken," i.e. treated with mineral acids, alkalies or salts in order to remove mucilaginous and solid or colloidal matter is disclaimed.

The invention is applicable to the production of super-drying oils from oils containing a high percentage of unsaturated glycerides, such as perilla, linseed, n'gart oil, walnut, poppy seed, soya bean, sunflower, candle nut, corn, cottonseed, salmon, cod-liver, sardine, menhaden. Linseed, perilla and walnut oils are specially suitable.

According to the invention such oils are subjected to a process of molecular distillation preferably at pressures in the neighbourhood of 0.001—0.0001 mm. mercury absolute pressure.

It is preferred, for reasons hereinafter stated, but not essential to the ultimate object of our invention, to first treat the oil by bubbling through it a stream of heated hydrogen, the purpose of which is to sweep out air and other gases contained in the oil, replacing them with hydrogen.

After this pretreatment of the oil, it is led into a de-gassing apparatus, which consists substantially of a chamber or chambers through which the oil passes in a thin film, and in contact with surfaces heated so as to cause the oil to obtain a temperature of approximately 100° C. The chamber or chambers are subject to evacuation by the use of a suitable vacuum pump so that the gas pressure within the chamber, during the passage of the oil, is reduced to and maintained at about 10 mm. of mercury absolute pressure. This treatment suffices to remove substantially all of the dissolved and/or occluded gases contained in the oil, and with such oils as linseed further serves to remove a considerable quantity of the substances responsible for the taste of the oil. We have also found it desirable to divide the degasser into two separate compartments, through which the oil passes in series, and in the first compartment to carry a lower temperature of the oil and a higher gas pressure, while in the second compartment a higher temperature is provided with a lower gas pressure.

From the degassing apparatus, the oil

[Price 1/-]

is conducted in closed pipes or tubes to a molecular distilling apparatus, substantially as described by U.S. Patent Specifications Nos. 1,925,559 and 1,942,858.

It is obvious that under the conditions obtaining in a molecular still, water vapour is effectively removed, since it would not be condensible under the conditions obtaining during the distillation. It is further obvious that the process involves the use of no reagents that would leave undesirable residues or by-products, the process being entirely physical.

With respect to the pretreatment with hydrogen, this, as previously pointed out, is not essential to the ultimate object of the invention, but serves to facilitate the holding of the required degree of vacuum within the still, since it quite effectively displaces gases of heavy molecular weight occurring in the crude oil, and due to the light weight of hydrogen it is more readily pumped out under conditions of high vacuum.

The free fatty acids and sterols collect as a distillate from the first stage of the still, and will vary in amount with the condition of the original oil. The "late" fraction constitutes a highly purified oil which finds a ready use as a super-drying oil for admixture with white lead in the preparation of paints and the like.

The residue from the still may either be put to any desirable use or rejected. A typical example of the fractions obtained, together with their character, from linseed oil, is given:

EXAMPLE.

Crude linseed oil is degassed according to recognised methods and is then passed through a molecular still, having a succession of surfaces exposed to successively high temperatures. Two fractions, 1 and 2 as below, are withdrawn at temperatures of 190° and 210° respectively, at pressures of residual gas not exceeding 5×10^{-3} and 10^{-3} mms. mercury, respectively. These two fractions are discarded since they contain the odorous and accessory substances. A third fraction, No. 3, as below, of twenty to twenty-five per cent. is withdrawn and a fourth fraction, No. 4 as below, of equal bulk. These fractions distil at temperatures ranging from 225° to 250° C. at pressures below 10^{-2} mm. mercury and preferably in the range of 0.5 to 3×10^{-3} mm. mercury. A residue of oil comprising ten to twenty per cent. of the original is rejected undistilled. The characteristics of the starting material and of the fractions referred to are best seen from the table set out below.

Linseed Oil.

	Fractions.					
	Original	1	2	3	4	Residue.
Iodine No. -	177.9	148.7	148.0	173.3	187.9	190
Saponification No. - -	192.5	171.8	163.1	183.3	194.2	184.1
Non-saponifiable matter - -	1.36	16.14	2.67	0.93	0.56	0.93
Free fatty acids as Oleic acid -	1.6	36.8	0.8	0.25	0.25	0.37
Unsaturated Fatty acid.	85.57	69.45	86.11	88.11	88.04	88.6

Fractions 1, 2 and 3 are discarded but fraction 4, which has an iodine value substantially higher than that of the initial material, is suitable for use as a super-drying oil.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

The process of distilling unbroken linseed and similar oils under conditions pertaining to molecular distillation, so as to obtain one or more "late" distillate fractions which are more unsaturated than the initial oil.

Dated this 19th day of January, 1938.

W. P. THOMPSON & CO.,
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Chartered Patent Agents.

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